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Setting characteristics of a resin infiltration system for incipient caries treatment

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Abstract: OBJECTIVES: This study investigated the curing efficiency, the extent of atmospheric oxygen inhibition to the polymerization reaction and the mechanical properties of a new resin-infiltration system for caries treatment. METHODS: The curing efficiency was estimated by measuring the percentage degree of cure (%DC) of thin resin films (h: 150 m, Ø: 5mm, n=3), 10min after 40s exposure to a quartz halogen bulb (750mW/cm2) light curing unit (Optilux 501, Demetron/Kerr, USA), in the absence of O2, by micro ATR-FTIR spectroscopy. The extent of O2 inhibition on resin film setting (width in m) was assessed by transmission optical microscopy on thin films (h: 150 m, Ø: 7mm, n=5) placed between two transparent cover slips and exposed to air from lateral sites. For each sample the extent of inhibition was measured at 5 different locations. The mechanical properties were tested employing Instrumented Indentation Testing according to ISO 14577:2002. Resin specimens (h: 2mm, Ø: 10mm, n=5) were prepared employing cylindrical teflon moulds enclosed in transparent matrix strips and glass slides as before. The measurements were performed employing a Vickers indenter attached to a universal hardness testing machine (ZHU2.5/Z2.5 plus test Xpert software, Zwick/Roell, Ulm, Germany). The parameters tested were Martens Hardness (MH), Vickers Hardness (VHN), Indentation Elastic Modulus (EIT) and elastic to total ratio of indentation work (IT). For all these tests, specimens of a conventional light-curing bonding resin (HB-Heliobond, Ivoclar-Vivadent, FL) prepared as above were used as control. Student t-test was used to identify statistically significant differences between the two materials in the parameters tested (a: 0.05). RESULTS: The results of the materials tested were: (a) [% DC]; IC: 57.4±1.5, HB: 59.8±2.4, (b) [Width of O2 inhibition/ m]; IC: 33.1±6.5, HB: 23.6±4.4, (c) [MH/N/mm(2)] IC; 116±16, HB: 261±35, (d) [VHN]; IC; 15.4±2.5, HB: 22.1±1.8, (e) [EIT/(GPa)]; IC; 2.3±0.4, HB: 7.5±0.5, and (g) [IT (%)] IC; 50.3±3.4, HB: 35.1±1.9. The IC presented no significant difference in terms of % DC, higher thickness of the inhibited layer, lower MH, VHN, EIT and greater IT values than HB. CONCLUSIONS AND CLINICAL SIGNIFICANT: The resin-infiltrating system for incipient caries treatment demonstrated the same curing efficiency with a conventional unfilled bonding resin, but exhibited higher extent of oxygen inhibition, lower hardness, lower elastic modulus and higher plastic to elastic indentation energy.

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Setting characteristics of a resin infiltration system for incipient caries treatment.

Short title: Setting characteristics of a resin infiltration system

Original research

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Key Words: caries, infiltration, ICON, mechanical properties, resin composites, oxygen inhibition

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Abstract

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N/mm^2] IC; 116±16, HB: 261±35. d) [VHN]; IC; 15.4±2.5, HB: 22.1±1.8. e) [E_{IT}/(GPa)]; IC; 2.3±0.4, HB: 7.5±0.5. and g) [η_{IT} (%)] IC; 50.3±3.4, HB: 35.1±1.9. The IC presented no significant difference in terms of % DC, higher thickness of the inhibited layer, lower MH, VHN, E_{IT} and greater η_{IT} values than HB. Conclusion: The resin-infiltrating system for incipient caries treatment demonstrated the same curing efficiency with a conventional unfilled bonding resin, but exhibited higher extent of oxygen inhibition, lower hardness, lower elastic modulus and higher plastic to elastic indentation energy.

Introduction

Active or arrested incipient caries lesions localized in enamel or in the external part of dentine, are clinically characterized by their compliance with the ICDAS scoring criteria (1). For the management of these types of lesions, several protocols have been introduced aiming to facilitate the natural reminerilization process of the lesion through the application of fluorides, calcium phosphates with peptides or amino acids, the enhancement of oral hygiene methods, the increase of local concentrations in minerals and the pH increase of the local micro-environment (2)

Caries infiltration (3) is a new micro-invasive treatment option for such cases, where enamel porosities attributed to mineral loss during demineralization are occluded by infiltration with a low-viscosity, light-cured resin, which a) penetrates the entire lesion extension, b) mechanically stabilizes the fragile, porous enamel structure and c) isolates microbia from their nutritious environment. This technique is mainly indicated for patients with low compliance to other management strategies, for lesions where aesthetics is a principal issue and in micro-cavitated lesions, with compromised plaque removal capacity.

The benefits of the resin infiltration technique have been clinically documented (4, 5) in cases of proximal and free-surface enamel lesions, which were efficiently infiltrated instead of undergoing more invasive restorative approaches. Recently, *in vitro* evidence of enhanced infiltrant penetration has been corroborated by *in situ* (6) and short-term clinical trials (7, 8) which have shown the effectiveness of resin infiltration in preventing further demineralization under cariogenic conditions. Although, several parameters of the resin infiltration technique have been investigated such as penetration capacity, etching procedure or physical properties of the material, (9-11) there is a lack of evidence about the setting characteristics of the films formed by the new material.

The aim of the present study was to investigate the curing efficiency, the extent of atmospheric oxygen inhibition to the polymerization reaction and the mechanical properties of a new resin-infiltration system for caries treatment. The null hypothesis was that the new system demonstrates improved properties when compared to a conventional bonding resin.

Materials and Methods

The composition of the caries infiltrating system and the conventional bonding resin used in the study are listed in Table 1.

a) Curing efficiency.

Resin specimens were fabricated using cylindrical Teflon molds (h:150μm, Ø:5mm, n=3) placed on microscopic glass slides covered by transparent cellulose strips. The molds were filled up with each material, covered with transparent strips and slides and photopolymerized for 40 s with a quartz halogen bulb light curing unit (Optilux 501, Demetron/Kerr, USA, standard mode at 650mW/cm^2). Following 10 min storage at 37^{o}C the specimens were removed from the molds and studied by micro-attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR). The base of each specimen was pressed against a 2x2 mm diamond reflective element of a single-reflection micro ATR attachment (Golden Gate, Specac, Slough, UK), equipped with ZnSe lenses, which was placed at the sample compartment of an FTIR spectrometer (Spectrum GX, Perkin-Elmer Corp., Bacon, UK) and spectra were taken under the following conditions: DGTS detector, 4000-650 cm^{-1} wave number range, 4 cm^{-1} resolution, 40 scans co-addition, 2 μm dept of analysis at 1000 cm^{-1} and 25 ±1 ^{0}C chamber temperature. Spectra of unset materials were taken under the same conditions by applying each material onto the horizontal sampling surface of the refractive element. The percentage degree of C=C bonds conversion (DC%) was calculated employing the two-band technique, by measuring the peak height ratios of C=C (1638 cm^{-1}) to aromatic C..C (1605 cm^{-1}) stretching vibrations for HB and the peak height ratios of C=C (1638 cm^{-1}) to ester C=O (1715 cm^{-1}) stretching vibrations for IC (12). All ratios were normalized vs the corresponding ratios of the unset controls and the resultant values were subtracted from 100 to calculate the DC% values.

b) Extent of oxygen inhibition.

The extent of O2 inhibition on resin film setting was assessed by transmission optical microscopy on thin liquid films (h:150μm, Ø: ~7mm, n=5) placed between two transparent cover slips (Figure 1). The resin films, during setting, were exposed to air from lateral sites. The films were exposed to the light-curing unit for 40 s and the width of the inhibited zone was measured on each sample immediately after setting at five different locations under 50X magnification employing an optical microscope (Leica 4000B, Leica Microsystems, Wetzlar, Germany) operated in transmission mode. The atmospheric conditions were room temperature 20 ±1^{0}C and humidy 55±5

%.

c) Mechanical properties.

The mechanical properties tested were determined employing Instrumented Indentation Testing (IIT) according to ISO 14557:2002 (13). Five resin specimens (h:2mm, Ø:10 mm, n=5) were prepared from each material employing cylindrical teflon molds enclosed in transparent matrix strips and glass slides as before. The specimens were light-cured as previously described and stored for 10 min at 37°C under dark and dry conditions. The measurements were performed employing a Vickers indenter attached to an IIT machine (ZHU2.5/Z2.5 plus test Xpert software, Zwick/Roell, Ulm, Germany) applied to the directly exposed surfaces with the indenter placed at 2 mm distance from the specimen margins under the following conditions: 1 kp load, 40x magnification (optical system), 2s load application, 0.5 mm/min load application speed, 0.1 mm/min load contact and removal speed. The parameters derived according to mathematical formulas provided by ISO 14557 were Vickers Hardness (VHN), Martens Hardness (MH), Indentation Elastic Modulus (E_{IT}) and elastic index defined as plastic to elastic ratio of indentation work (η_{IT}). Vickers hardness was defined by measuring the diagonal length of indentation with the optical system while the rest properties determined based on force-indentation depth curve.

d) Statistical analysis

Student t-test was used to identify statistically significant differences between the two materials in the parameters tested, at a 95% confidence level (a=0.05), employing the SPSS v 19.0 software package.

Results

The DC% values measured are presented on Table 2. They showed no statistically significant difference between the two materials ($p>0.05$).

Fig. 2 shows a representative image of the extent of O_2 inhibition zone in IC. The results of the extent of O_2 inhibition (Table 2) demonstrated a statistically significant greater O_2 inhibited layer in IC in comparison with HB ($p<0.05$).

Representative loading-unloading force - indentation depth graphs for IC and HB are illustrated in Fig.3. The results of the mechanical properties as determined by IIT are summarized in Table 2. HB demonstrated greater MH, VHN, E_{IT} and lower η_{IT} values than IC. All mean differences were statistically significant ($p<0.05$).

Discussion

The infiltration of caries lesions with low viscosity dimethacrylate resins is considered as an important treatment option for incipient caries lesions (7). In contrast to the conventional sealing concept of occlusal pits and fissures, caries infiltrating agents should penetrate into the entire porous lesion body, without occupying free surface volume. The latter is important to manage the space problems faced in marginal applications.

HB, a traditional light-curing bonding resin, has the capacity to penetrate into early enamel lesions (14, 15), efficiently bond to acid-etched enamel (16-18) and effectively seal artificial lesions (19, 20). For all these reasons, HB was chosen as a control material. Based on the results of the present study, IC showed inferior properties than HB, except from DC%, were no statistically significant difference was found between the two materials. Therefore, the null hypothesis should be rejected.

The FTIR analysis offers a direct and reliableapproach to evaluate the %DC in dental resin composites. The results of DC% measurements imply that both materials produced a highly saturated polymer network, as they contain in high percentage TEGDMA, a low viscosity, flexible and highly reactive monomer. It has long been recognized that increased DC% of resin composites is associated with improved mechanical and chemical properties, color stability and biocompatibility (20). However, since the strength of the unfilled polymers tested is mainly dependent on monomer composition, bulk monomers with a rigid aromatic backbone, like BisGMA in HB, increase the strength of the set material. It has been well documented that BisGMA polymers show higher flexural strength and elastic modulus than TEGDMA polymers regardless the higher DC% obtained from the latter (21). The higher elastic modulus of the BisGMA containing HB, as confirmed in the present study, may explain the increased hardness in comparison with the TEGDMA-rich IC. Moreover the higher elastic index (ηIT) found in IC clearly indicates that its polymer network is less capable of plastic deformation compared to HB.

The use of unfilled TEGDMA-based resin in IC is apparently based on the low molecular weight and viscosity of the monomer, in comparison with other aromatic

(BisGMA, BisPMA, BisEMA etc) or non aromatic dimethacrylate monomers (UEDMA, DUDMA), with higher molecular weight and viscosity, used in dental resins technology. Therefore, by using TEGDMA, penetration into the porous incipient carious lesions of a hydrophobic material, is enhanced. However, it has been shown that TEGDMA is selectively released from homo- or co-polymers with rigid aromatic dimethacrylate monomers after immersion in water or in polar solvents (22), implying a polymer network prone to chemical degradation. This may explain the findings of a recent study, where the protective effect of HB exerted on enamel exposed to an acidic environment, was higher than that of IC (23).

Most dental resins used for conservative and preventive treatments are polymerized intraorally by free radical polymerization. Environmental oxygen is a powerful inhibitor of this setting mechanism by reacting with free radicals produced and forming unreactive peroxy radicals, which retard or even terminate polymerization, creating thus a poorly polymerized or totally inhibited resin surface layer (24). It has reported that the oxygen inhibition layer thickness for dental resins varies from 4 to 60 μm (25, 26), dependent on the initiation mode (chemically or light curing), initiator concentration, monomer viscosity, filler size and loading, and resin-filler interactions (27). Viscous monomers and presence of fillers provide a diffusion barrier to oxygen, whereas fast and massive production of free radicals, like in light-curing, favors surface setting and reduces the extent of oxygen inhibition. Assuming an equally efficient light-curing mechanism in both the unfilled materials tested in the present study, the difference found in the extent of oxygen inhibition should be exclusively attributed to monomer composition. The main reason is that the low viscosity TEGMA resin in IC cannot inhibit oxygen diffusion, as effectively as the more viscous BisGMA monomer in HB.

The extent of oxygen inhibition has a critical impact on the setting of thin resinous films in the absence of coating, like the treatment of smooth surfaces with IC. Films less than 30 μm cured in air may not set. Although the infiltrated part in etched enamel, may be partially protected from oxygen diffusion due to the etching pattern and the resultant enamel prism protrusions, the superficial layer left is completely unshielded. Consequently, part of enamel surface, including the exposed prism protrusions, may be exposed intraorally following rinsing. The incomplete surface sealing may explain the better protective effect to enamel demineralization exerted by HB in comparison with IC (23).

The comparison of mechanical properties clearly shown that HB has better mechanical properties, combining also a more plastically deformed matrix as indicated by its lower elastic index (28). As previously discussed, the presence of the rigid BisGMA monomer in HB may explain the higher values of hardness and E_{IT} recorded. Another explanation for the differences in surface hardness should be the differences in crosslink density, as well as HB is a co-monomer mixture and IC exhibits homopolymerisation. Although Vickers hardness has been extensively used to characterize the hardness of dental materials, its accuracy is influenced by the resolution of optical system used to measure the diameter of indentation, the operator's perception and by the elastic recovery of material around indentation (29) Contrary, Martens hardness is free of aforementioned complications as it is measured directly from the force indentation depth curve data (18, 29). Mechanical strength of protective resin coatings applied to smooth enamel surfaces is an important issue, considering that all dental polymers are subjected to intraoral plasticization and toothbrush abrasion. The harder HB may anticipate improved protection. This may explain the improved enamel protection obtained when IC was combined with conventional bonding resins based on BisGMA/TEGDMA co-monomers (30).

Conclusions

The resin-infiltrating system for incipient caries treatment demonstrated the same curing efficiency with a conventional unfilled bonding resin, but exhibited higher extent of oxygen inhibition, lower hardness, lower elastic modulus and higher plastic to elastic indentation energy. The inferior properties of IC may affect the sealing capacity, mechanical strength and durability of the intraorally exposed part of the infiltrating coating.

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Table 1. The products tested

PRODUCT/LOT	CODE	COMPOSITION (wt%)*	MANUFACTURER
ICON 621818	IC	TEGDMA-based resin, initiators and stabilizers	DMG, Hamburg, Germany
Heliobond S46831	HB	BisGMA (59.5), TEGDMA (39.7), initiators and stabilizers (0.8)	Ivoclar Vivadent,Schaan, Liechtenstein

*According to manufacturers' information

Table 2: The results of DC%, extent of O_2 inhibition and mechanical properties tested (means and standard deviations in parentheses). Different superscripts indicate statistically significant differences between the products per property ($p<0.05$).

Product	**DC%**	**O_2 inhibition (µm)**	**MH (N/mm^2)**	**VHN_1**	**E_{IT} (GPa)**	**η_{IT} (%)**
IC	57,4 (1,5)[a]	33,1 (6,5)[a]	116 (16)[a]	15,4 (2,5)[a]	2,3 (0,4)[a]	50,3 (3,4)[a]
HB	59,8 (2,4)[a]	23,6 (4,4)[b]	261 (35)[b]	22,1 (1,8)[b]	7,5 (0,5)[b]	35,1 (1,9)[b]

Fig 1

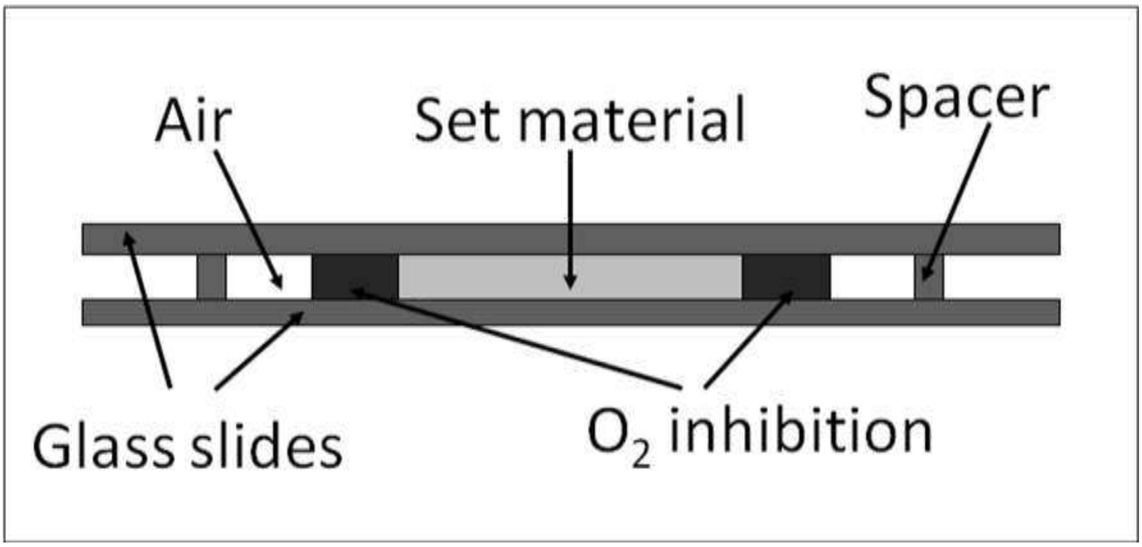


Fig. 2

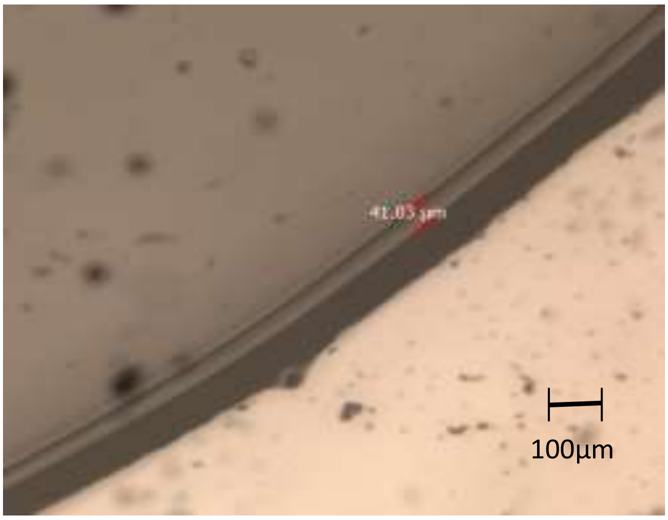
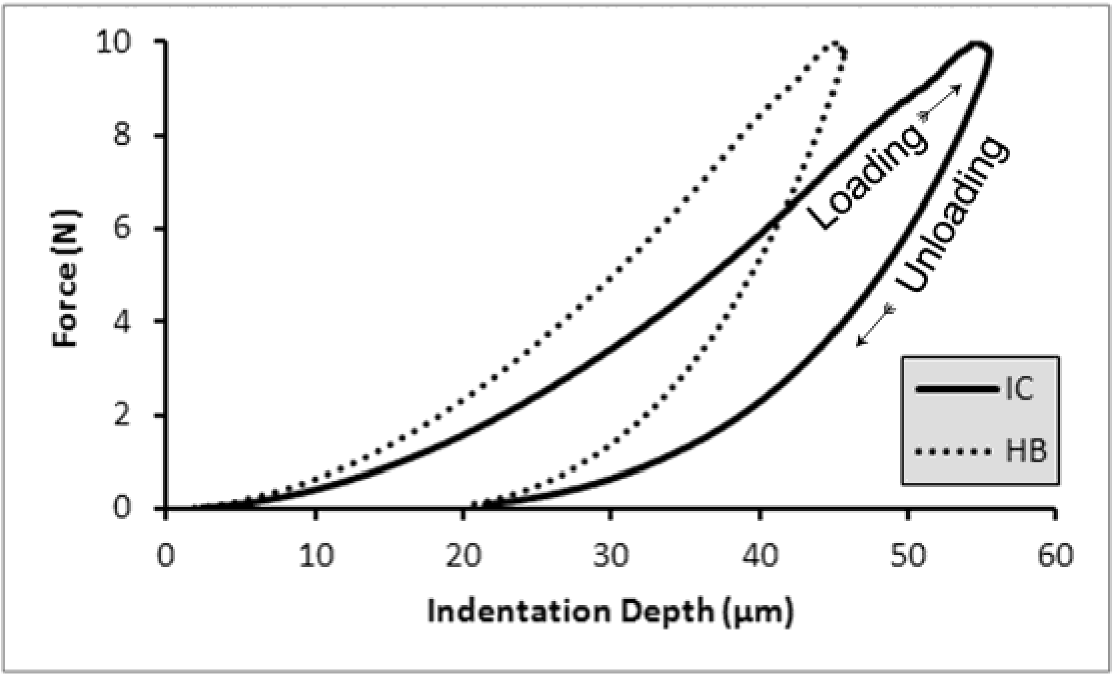


Fig. 3



Force (N)
10
8
6
4
2
0
0
10
20
30
40
50
60
Indentation Depth (µm)
Loading
Unloading
IC
HB

Legends

Figure 1. Schematic representation of the set up used for the assessment of the extent of O_2 inhibition.

Figure 2. Photomicrograph of the O_2 inhibited zone of IC infiltrant resin (transmitted light, 50X magnification).

Figure 3. Representative force indentations depth curves of both materials included in the study. The loading-unloading direction is pointed by the arrows for one of the products tested.